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**SUPERCritical EXTRACTION
OF POPLAR WOOD
IN A CONTINUOUS SYSTEM**

RENEWABLE ENERGY BRANCH

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ABSTRACT

Continuous supercritical extraction of poplar wood at 360°C and 20 MPa pressure and a similar extraction with hydrogen addition (Runs 1 and 2) were successfully carried out using the B.C. Research continuous coal liquefaction unit.

Dependable delivery of methanol wood slurry to the high pressure reactor was a serious problem during the initial stage of this investigation. It was solved by streamlining feed lines and diluting the slurry to 5 weight percent wood.

The respective yields of liquid products isolated in Runs 1 and 2 were 70 and 51% based on dry wood while the yields of gas were 6 and 31% and water were 3 & 1%. Conversions of 93-94% based upon ash balances showed no significant differences due to hydrogen addition. Limited material balance data of 94 and 83% for Runs 1 and 2 were insufficient for accurate economic evaluation of the process.

The products of Runs 1 and 2 showed significant chemical and physical differences. The hydrogen: carbon ratio decreased from 1.52 to 1.44 with hydrogen addition in Run 2, while the viscosity increased from 43 to 61 cP and the methoxyl content decreased from 8.9 to 7.3%. Thermogravimetric data showed that hydrogen addition decreased volatility in the 100-400°C range. However, neither densities (1.1 g/cm^3), non-distillable residues (8.6%) nor heating values (6.4 kcal/g) were significantly affected by hydrogen addition.

Résumé

On a utilisé avec succès le système de liquéfaction du charbon de la B.C. Research pour l'extraction continue de bois de peuplier dans un milieu surcritique à 360°C et à 20 MPa, sans et avec ajout d'hydrogène (Essais 1 et 2).

Au cours du stade initial de l'étude, l'alimentation du réacteur à haute pression en pâte de méthanol et de bois a causé de sérieux problèmes. Ces problèmes ont toutefois été résolus grâce à l'alignement des conduites d'alimentation et à la dilution de la pâte à 5 % de bois en poids.

Lors des deux essais, la production de liquides s'est établie respectivement à 70 % et à 51 % par rapport au bois sec alors que la production de gaz se chiffrait à 6 % et à 31 % et celle d'eau à 3 % et 1 %. Les conversions de 93 % à 94 % fondées sur le bilan des cendres n'ont révélé aucune différence notable en fonction de l'ajout d'hydrogène. Les données, limitées à 94 % et 83 % pour le bilan de matières, n'ont pas permis une évaluation économique exacte du processus.

Les produits issus des deux essais différaient beaucoup chimiquement et physiquement. Lors du deuxième essai, avec l'ajout d'hydrogène, la proportion d'hydrogène par rapport au carbone a diminué de 1,52 à 1,44 alors que la viscosité a augmenté de 43 à 61 cP et que la teneur en méthoxyle a diminué de 8,9 % à 7,3 %. Les données thermogravimétriques ont démontré qu'à des températures variant entre 100 et 400°C, l'ajout d'hydrogène faisait diminuer la volatilité. Toutefois, ni les densités ($1,1 \text{ g/cm}^3$), ni les résidus distillables (8,6 %), ni le pouvoir calorifique (6,4 kcal/g) n'ont été sensiblement touchés par l'ajout d'hydrogène.

1. OBJECTIVES

The main objective of this work was to show that continuous extraction of wood with supercritical fluids could be achieved with equipment designed for continuous liquefaction of coal.

A secondary objective was to prepare poplar wood oils by continuous supercritical extraction for comparison with products obtained by other poplar wood liquefaction processes under investigation in Canada.

2. BACKGROUND

Previous work at B.C. Research, under ENFOR Project C-51, successfully demonstrated that supercritical fluids could liquefy wood to produce oil. Organic solvents such as acetone and methanol, heated above their critical temperatures and compressed above their critical pressures, gave high oil yields from wood. Yields of 70% were obtained using either supercritical acetone or methanol at 360°C and 28 MPa, in a batch system. Acid pretreated wood also gave these yields, but under milder conditions (260°C, 10MPa).

Despite these encouraging results it was realised that on a commercial scale, a continuous extraction process would be required. Batch loading of extraction vessels and time required to heat batches to reaction temperature were considered inefficient and uneconomic.

It was hoped therefore that the B.C. Research continuous coal liquefaction unit shown in Figure 1 could be adapted to wood liquefaction using supercritical fluid extractions with only minor modifications.

3. EXPERIMENTAL METHODS

3.1 MATERIALS

The poplar wood was supplied by Forintek Canada Corp. in chip form.

Analytical grade methanol from BDH was used in the experiments.

3.2 EQUIPMENT

3.2.1 B.C. Research Continuous Coal Liquefaction Unit

The continuous extractions were carried out in the B.C. Research continuous coal liquefaction unit, a schematic of which is shown in Figure 1. The unit consists of a feeding system, a reactor and a product recovery unit.

The feeding system delivers a coal slurry and hydrogen at high pressure to the reactor. Coal slurry is stored in a stirred metal container from which it passes via a recirculation pump into a high pressure metering pump. The recirculation pump manufactured by Robin & Myers (Moyno Pump Model 6M1-CDF) helps to keep slurry from settling and provides some inlet pressure for the metering pump. The metering pump is a Bran and Lubbe type NP-32 dual head positive displacement pump with 5 mm diameter plungers and triple ball check valves, capable of a maximum delivery of 2.76 L/hr per head at maximum pressure 40 MPa.

Hydrogen is compressed from the standard (13 MPa) cylinders into high pressure accumulators by a two stage gas compressor (Pressure Products Industries Model Q-047028). Hydrogen and the slurry are introduced to the bottom of the reactor. Hydrogen rate is measured by a mass flow meter (Hastings Model Nall-50KGX) and the slurry feed rate is determined by the slurry tank weight loss over the run duration period.

The reactor is a 2 L stainless steel autoclave (Autoclave Engineers, Inc.) with a magnetically driven stirrer which is heated by a controlled external electric furnace.

The product recovery system consists of heavy and light end receivers, a gas flow control valve and a gas meter.

Non-stirred 2 L autoclaves, similar to that used as the reactor, serve as heavy end receivers and non-stirred 300 mL autoclaves, also from Autoclave Engineers, are used as light ends receivers. The heavy end receivers can be optionally heated by controlled external electrical furnaces.

During a coal liquefaction run one heavy end receiver and one light end receiver are on stream while the other pair of receivers are depressurized for the product removal.

Hydrogen together with the product gas exit through a control valve. The exit gas rate is measured in a rotameter and its volume in a dry gas meter.

3.2.2 The Modified Feed System

To accomodate wood slurries and avoid plugging the original continuous coal liquefaction unit, feed system was modified in the following areas:

- sharp elbows were replaced with smooth bends
- the orifices of all valves were increased
- slurry consistency was reduced from 30 weight percent used in coal liquefaction to 5 weight percent

3.2.3 The Reactor

The only change to the reactor used for continuous coal liquefaction was the incorporation of a steel wool plug in the exit port in order to minimize solids carryover into the receiver. A dip tube reaching to the bottom of the reactor was used for the introduction of hydrogen in Run 2.

3.2.4 The Modified Product Recovery System

The continuous coal liquefaction reactor was attached to a water cooled condenser to condense both the methanol solvent and wood liquefaction products in a large receiver. [All complex equipment used to recover coal liquefaction products was bypassed in this project]. Non-condensable products were passed through an ice cooled trap and finally into a plastic gas collection bag (Run 1) or a dry gas meter (Run 2).

3.3 ANALYSES .

Proximate analyses of wood and residue were done on a Du-Pont 1090 Thermogravimetric Analyser (TGA).

Gases were analyzed for hydrogen, methane, carbon monoxide, carbon dioxide, oxygen and nitrogen by gas chromatography using a Hewlett Packard Model 5700A gas chromatograph on a Porapak Q column.

The water contents of liquid products were determined by Econotech Services Ltd., New Westminster, B.C. using the Karl Fischer method and elemental compositions were estimated by Canadian Microanalytical Laboratories Ltd., Vancouver, B.C.

Heating values of the liquids were determined in a Parr adiabatic calorimeter and viscosities in a Penske Martin capillary viscometer. The feed wood and the products were analyzed for methoxy groups ($(\text{CH}_3\text{-O-})$) using the ASTM Standard Method D 1166-60.

3.4 SLURRY PREPARATION

The only procedure capable of producing a wood slurry which could be pumped into the high pressure reaction vessel following streamlining described in 3.2.2 involved the following steps:

- Wiley mill grinding of poplar wood chips
- Pullman mill grinding of the Wiley mill product in a closed cycle with partial air classification
- Wet screening of the Pullman mill material in methanol through a 140 mesh screen
- Adjusting the -140 mesh particulates to a slurry strength of 5% by weight in methanol.

3.5 OPERATION OF THE MODIFIED CONTINUOUS COAL LIQUEFACTION EQUIPMENT

3.5.1 Liquefaction

After the reactor had been charged with 450 g of the 5 weight percent wood slurry in methanol it was pressure tested with nitrogen and then vented. It was then flushed with hydrogen when this gas was added to the reactor during Run 2. The temperature of the reactor was then raised to 360°C and the metering pump started. Settling of wood in the slurry feed was prevented by keeping the recirculating pump operating continuously. The pressure in the reactor was controlled manually at 20 MPa. Methanol together with extract and some carry over solids were condensed and collected in a large receiver. Gases were either collected in a plastic bag (Run 1) or metered in a dry gas meter and sampled at regular intervals (Run 2). At the end of the run the metering pump and hydrogen flow (in Run 2) were stopped and the exit valve from the reactor closed. The reactor was then cooled with compressed air and the contents removed, after pressure had been released.

3.5.2 Product Separation

The condensate and the material removed from the reactor after cooling were weighed and filtered and the liquids were combined. The solids were dried in a vacuum oven at 40°C and weighed.

The liquid products were recovered from the condensate following distillation of the solvent and water. The bulk of the methanol was removed on a short distillation column and separation was completed on a 200 plate spinning band distillation column.

4. STAFF

Mr. Josef Jezko was the Project Leader for the continuation of ENFOR Project No. C51. Dr. John Howard was Program Manager. Ms. Bridle Bennett and Ms. Karen Totland provided technical assistance in the extraction and analysis of products.

5. ACKNOWLEDGEMENTS

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6. RESULTS AND DISCUSSION

6.1 GENERAL

Table 1 summarizes the proximate analyses of the poplar wood feed, carry over solids and the residue. The last column shows the total conversion calculated from ash data using the Neavel's formula (1):

$$\text{Conversion (Wt. \%)} = \frac{(A-a)10^4}{A(100-a)}$$

where A = % ash in dry residue

a = % ash in dry feed

Table 2 lists the experimental conditions for both continuous runs and Table 3 summarizes material balance data.

The results of gas analyses are presented in Table 4. Elemental composition of feed wood and of liquid products are shown in Table 5. Some physical properties of liquid products are given in Table 6. Results of the methoxy determinations for the feed and products are summarized in Table 7.

6.2 MATERIAL BALANCES

As shown in Table 3, the 94% material balance achieved in Run 1 is better than that the 83% achieved in Run 2. The poor material balance of Run 2 is a consequence of several restarts with resulting loss of slurry and/or methanol. The 94% material balance in Run 1 is satisfactory considering the short duration of the run and the fact that this was the first time wood had been successfully extracted using the continuous coal liquefaction unit.

Due to the relatively short duration of the runs and less than perfect material balances, any economic evaluation of the process based on these data at this stage would be of little value.

6.3 CONVERSION

Two kinds of residual solids were collected after the continuous extraction runs. These were solids which accumulated in the reactor during the run, and solids which were carried over with the supercritical fluid and products into the receiver. The latter amounted to approximately 25% of total residue.

The total conversion results shown in the last column of Table 1 indicate that both the carry over and accumulated solids, were largely (85-94%) converted to products. While there is no significant difference between Runs 1 and 2 in conversion of the material accumulated in the reactor (94 and 93% respectively), the carry-over material in Run 1 shows slightly higher conversion (89%) than the carry-over material in Run 2 (85%).

This difference in conversion is believed to be due to the combined effects of hydrogen addition in Run 2, which dilutes the supercritical methanol extraction medium, and the faster slurry feed rate in this run. The latter decreases the residence time of smallest wood particles in the reactor and such particles subsequently get carried over into the receiver.

In general, however, the conversion results indicate that the addition of hydrogen in Run 2 did not have a significant effect on total conversion of wood to liquefaction products.

6.4 YIELDS

There are large differences in the production of liquids and gases between the Runs 1 and 2 (Table 3). While Run 1 resulted in yields of 70% oil and 6% gases based on dry wood, in Run 2, with addition of hydrogen, oil yield dropped to 51%, but gas yield increased to 31%. It would appear that pyrolyzation reactions occurred to a higher degree in Run 2 in the presence of hydrogen than in Run 1 in its absence. The greater degree of pyrolyzation in Run 2 could possibly be attributed to a faster heat transfer with hydrogen

introduction. Improved heat transfer in hydrogen would conceivably permit use of lower extraction temperatures without reducing yield in any future extractions. The amount of water produced during the extraction was higher in Run 1 (3%) than in Run 2 (1%) indicating that the hydrogen addition suppresses water formation reactions.

6.5 LIQUID PRODUCT QUALITY

The hydrogen to carbon atomic ratio (H/C) shown in Table 5 for Run 1 liquid is higher than that of product recovered in Run 2 (1.52 and 1.44 respectively). This is despite the fact that Run 2 was carried out with the addition of hydrogen. This unexpected observation is probably due to the more vigorous pyrolyzation reactions occurring during Run 2. Here the faster heat transfer in the presence of hydrogen leads to some carbonization of the product.

While the non-distillable residues and heating values of both liquids do not significantly differ from each other (Table 6), the viscosity of liquid obtained in Run 2 is higher than that obtained in Run 1 (61 vs. 43 centipoises). This suggests that liquid produced in Run 2 contains a greater amount of higher molecular weight material than does liquid isolated in Run 1.

The comparative weight loss curves of both liquid products as determined by thermogravimetry (TGA) shown in Figure 2, indicate that middle range volatility (ca. 100-400°C) for liquid produced in Run 1 is slightly higher than that for liquid produced in Run 2.

Results of methoxy determinations shown in Table 7 indicate that some methoxylation took place during both runs. Most probably this was the result of supercritical methanol

reacting with alcohols and phenols produced during extraction. While there are more methoxy groups in the oil produced in Run 2 than in the oil produced in Run 1 (14.4 vs. 12.8 wt. %), when expressed on the basis of dry wood feed, the opposite is true (7.3 % vs. 8.9% respectively). These results indicate that the addition of hydrogen in Run 2 suppresses methoxylation reactions. If it is assumed that all the increase in methoxy group content is due to methanol reacting with the product, calculations show that only 0.2 weight percent of feed methanol is consumed by this reaction in Run 1 and 0.1 weight percent in Run 2.

7. CONCLUSIONS

The continuous coal liquefaction unit at B.C. Research can be adapted to supercritical methanol extraction of poplar wood at 360°C and 20 MPa by making the following modifications.

- Reducing the wood particle size in the methanol slurry feed to less than 106 microns (140 mesh).
- Reducing the slurry concentration to 5 weight percent
- Streamlining all feed lines
- Using large orifice check valves.

Yields of liquid products based on dry wood obtained in the modified continuous coal extraction unit are 70% with no hydrogen addition and 51% in the presence of this gas. The yield with no hydrogen addition is comparable to that obtained in a batch extraction system under similar conditions of pressure and temperature.

There are no advantages associated with hydrogen addition when the extraction is performed at 360°C and 20 MPa, both yield and product quality are adversely affected by resulting increased pyrolytic reactions. However, the increased heat transfer rate in the presence of hydrogen may allow future extractions to be carried out at lower temperatures without suffering yield or quality penalties.

8. REFERENCE

1. Neavel, R.C. FUEL, 1976, 55, p. 237.

TABLE 1
PROXIMATE ANALYSES OF FEED, CARRY OVER SOLIDS AND RESIDUE
TOTAL CONVERSION FROM ASH BALANCE

RUN	MATERIAL	AS ANALYZED (%)				DRY (%)			CONVERSION wt. %
		H ₂ O	VM	FC	Ash	VM	FC	Ash	
-	Feed	4.55	84.70	9.80	1.15	88.74	10.06	1.20	-
	Carry Over Solids	2.36	55.25	32.44	9.73	56.59	33.45	9.97	89
1	Residue	2.11	43.37	37.77	16.59	44.30	38.75	16.95	94
	Carry Over Solids	1.98	54.71	35.75	7.53	55.82	36.50	7.68	85
2	Residue	1.23	33.02	51.48	14.19	33.43	52.20	14.37	93

VM - Volatile matter.
FC - Fixed carbon.

TABLE 2
EXPERIMENTAL CONDITIONS FOR CONTINUOUS
SUPERCRITICAL EXTRACTION OF POPLAR WOOD
IN METHANOL

	Run 1	Run 2
Temperature	360°C	360°C
Pressure	20 MPa (3000 psi)	20 MPa
Slurry Density	5.0% wt.	5.0% wt.
Feed Rate (Slurry)	1.2 kg/hr	1.55 kg/hr
Feed Rate (Wood)	59 g/hr	77.5 g/hr
Hydrogen Rate	-	25 L/hr (average)
Run Duration	2 hr, 5 min	3 hr, 30 min
Initial Amount of Slurry in Reactor	450 g*	450 g

* Methanol only.

TABLE 3
SUMMARY OF RESULTS OF SUPERCRITICAL EXTRACTION
OF POPLAR WOOD IN METHANOL

	Run 1	Run 2
FEED (g):		
Methanol	2791	5585
Wood	123	294
Hydrogen	<u>-</u>	<u>8</u>
TOTAL	2914	5887
PRODUCTS (g):		
Methanol	2642	4592
Solids	7	21
Liquids	91	160
Gases	<u>7</u>	<u>95</u>
TOTAL	2747	4868
TOTAL MATERIAL RECOVERY (%)	94	83
YIELDS (wt. % dry wood):		
Solids	6	7
Oil	70	51
Water*	3	1
Gases*	<u>6</u>	<u>31</u>
TOTAL	85	90

* Does not include water in feed and introduced hydrogen.

TABLE 4
ANALYSES OF PRODUCT GASES (Vol. %)

Run	H ₂	CH ₄	CO	CO ₂	N ₂ *	O ₂ *
1	17.02	5.44	12.04	12.02	42.15	11.32
2 (average)	61.23**	5.01	12.03	18.61	2.67	0.51

* Air introduced in sampling.

** Includes % hydrogen added.

TABLE 5
ELEMENTAL COMPOSITION OF FEED WOOD
AND OF PRODUCT LIQUIDS

	AS ANALYZED (wt. %)				WATER FREE (wt. %)				H/C*
	C	H	N	O**	C	H	N	O	
Feed Wood	46.59	6.10	0.29	47.02	48.81	5.86	0.30	45.03	1.44
Run 1 Liquid	56.09	8.20	0.39	35.32	62.12	7.88	0.43	29.57	1.52
Run 2 Liquid	58.90	8.18	0.55	32.37	65.59	7.85	0.61	25.45	1.44

* H/C - Hydrogen to carbon atomic ratio.

** O - Oxygen by difference.

Table 6
SOME PHYSICAL PROPERTIES OF PRODUCT LIQUIDS

	NON-DISTILLABLE RESIDUE* (+525°C) wt. %	HEATING VALUE cal/g	VISCOSITY CENTIPOISE	SPECIFIC GRAVITY g/cm ³
Run 1	8.61	6424	43	1.1
Run 2	8.64	6461	61	1.1

* Material boiling above 525°C as determined by thermogravimetric analysis.

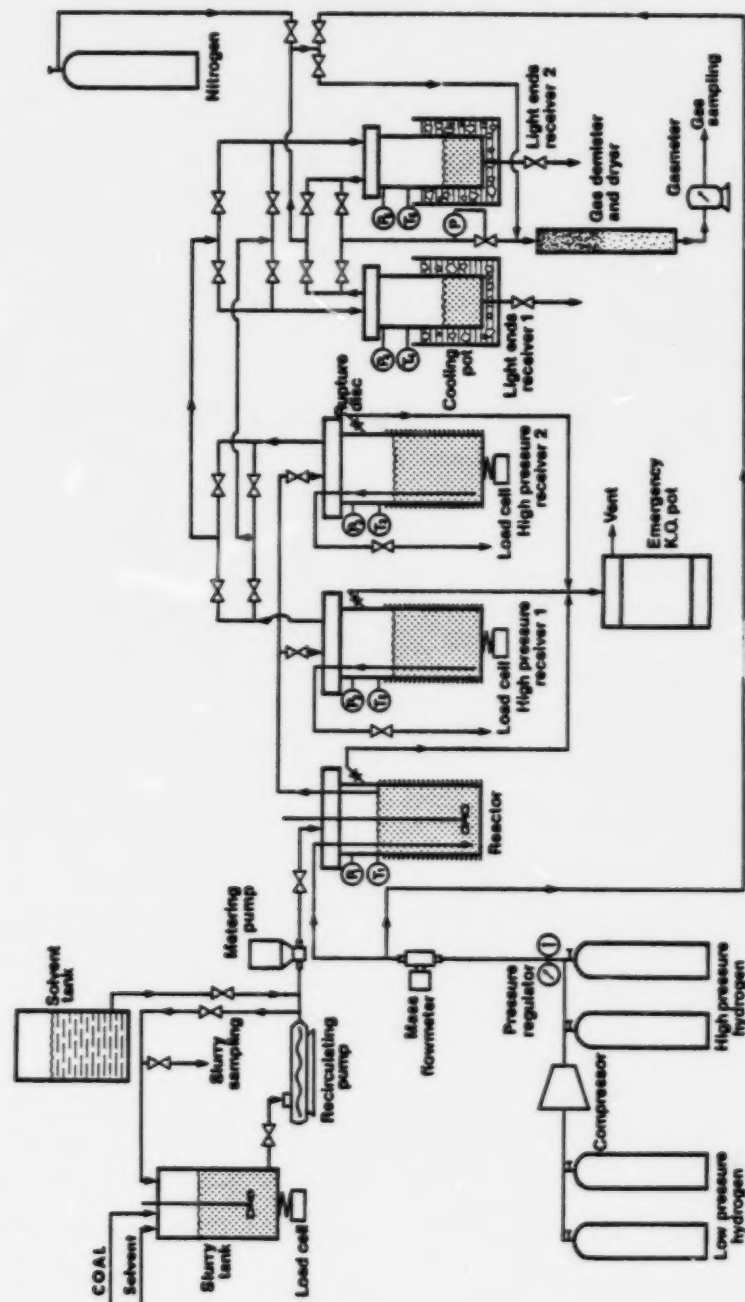
TABLE 7
METHOXY GROUP (CH₃-O-) CONTENT

		WT. % (WATER FREE)	WT. % OF DRY WOOD IN TOTAL FEED
	Feed Wood	4.9	
Run 1	011	12.8	8.9
	Carryover Solids	4.4	0.1
	Residue	2.4	0.1
Run 2	011	14.4	7.3
	Carryover Solids	5.0	0.1
	Residue	1.9	0.1

FIGURE 1

BC RESEARCH CONTINUOUS COAL LIQUEFACTION UNIT

Detailed Flow Diagram



Sample: SCE WOOD RUN #2 PRODUCT

Size: 81.77 mg

Rate: 20 DEG/MIN TO 525

Program: TGA Analysis V1.0

FIGURE 2.

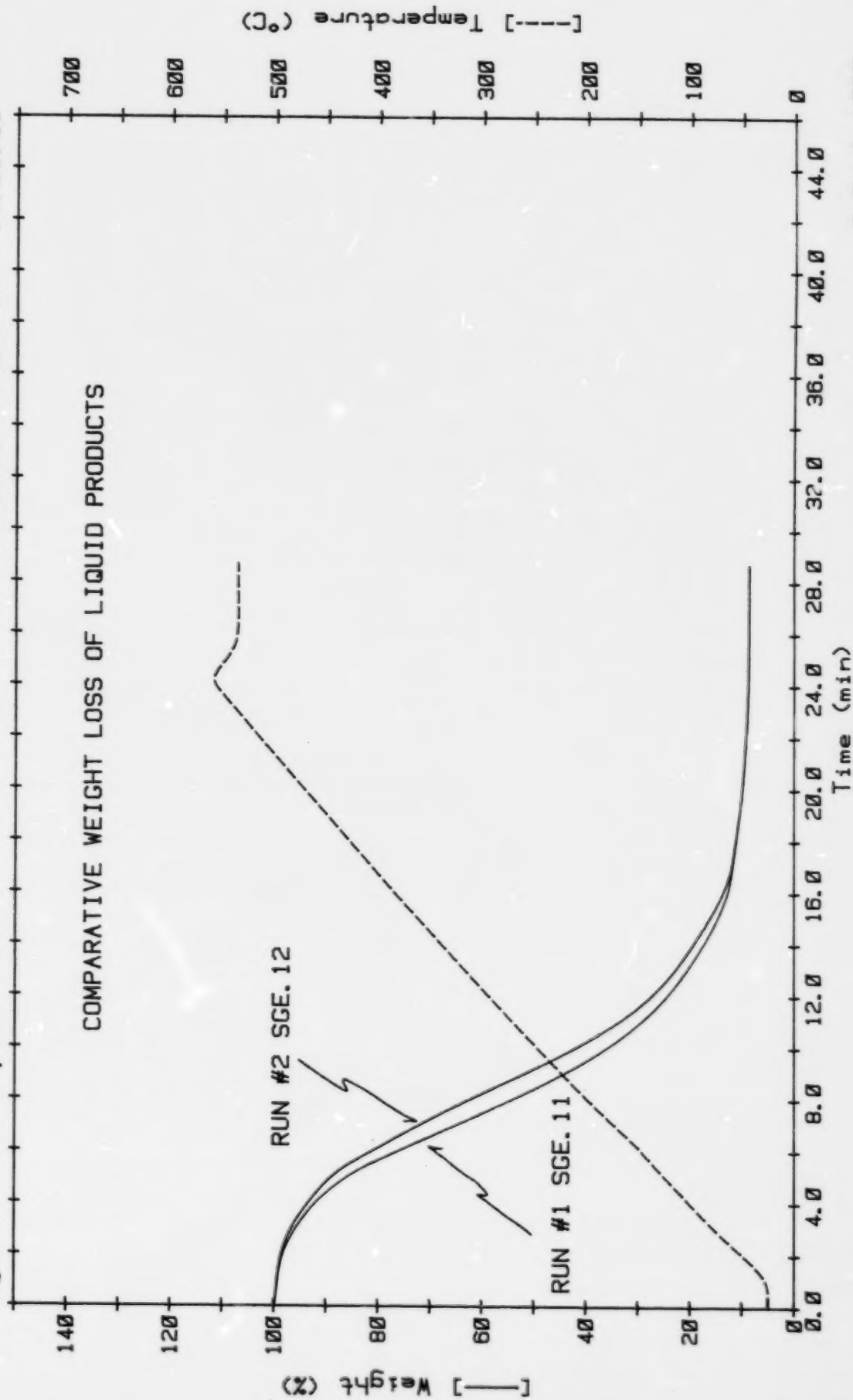
TGA

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File: SGE.12 PROX ANALYSIS XVI

Operator: ERIN

Plotted: 13-Jun-84 11:01:31



SUPERCritical EXTRACTION OF POPLAR WOOD
IN A CONTINUOUS SYSTEM

ABSTRACT OF THE ABSTRACT

A Tear Sheet Annotation

A modified continuous coal liquefaction unit was used to liquefy wood in 70% yield using supercritical methanol at 360°C and 20 MPa.